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Indian Standard

BORIC ACID FOR COSMETIC INDUSTRY — SPECIFICATION

(Fifth Revision)

भारतीय मानक

प्रसाधन उद्योग के लिए बोरिक एसिड — विशिष्टि

(पाँचवा पुनरीक्षण)

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 3

Cosmetics Sectional Committee, PCD 19

FOREWORD

This Indian Standard (Fifth Revision) was adopted by the Bureau of Indian Standards on 5 March 1990, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Boric acid is used as a preservative in cosmetic formulations. It finds use in aftershave preparations, bath preparations, deodorants, mouth washes, in nail bleaches and in skin powders.

In this revision, two new requirements, namely, fineness and sulphates have been included.

This standard covers requirements of boric acid for cosmetic industry only. Boric acid for non cosmetic use has been covered by a separate standard, IS 10116 : 1982.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shallbe rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

BORIC ACID FOR COSMETIC INDUSTRY — SPECIFICATION

(Fifth Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for boric acid for cosmetic industry.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
265 : 1987	Hydrochloric acid (*third revision*)
266 : 1977	Sulphuric acid (*second revision*)
323 : 1959	Rectified spirit (*first revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
2088 : 1983	Methods for determination of arsenic (*second revision*)
4905 : 1968	Methods for random sampling
10116 : 1982	Boric acid

3 REQUIREMENTS

3.1 Description

3.1.1 The material shall be in the form of a fine white powder or crystals or flakes, free from odour, dirt, foreign matter and visible impurities.

3.2 The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of the Table 1.

4 SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity, shall be as prescribed in Annex B.

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed in suitable containers of appropriate capacity, so as to ensure satisfactory stability and exclude contamination by extraneous matter, or in a manner as agreed to between the purchaser and the supplier.

5.2 Marking

5.2.1 The containers shall be securely closed and marked with the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Batch number in code or otherwise;
d) Date of manufacture; and
e) Gross, net and tare mass.

5.2.1.1 The containers may also be marked with the Standard Mark, details of which may be obtained from the Bureau of Indian Standards.

Table 1 Requirements for Boric Acid for Cosmetic Industry

(*Clauses* 3.2 *and* B-5.2)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Annex A)
(1)	(2)	(3)	(4)
i)	Boric acid (as H_3BO_3), percent by mass, *Min*	99·5—100·5	A-2
ii)	Arsenic (as As_2O_3), ppm, *Max*	10	A-3
iii)	Heavy metals (as Pb), ppm *Max*	20	A-4
iv)	Loss on drying, percent by mass, *Max*	0·5	A-5
v)	Iron, percent by mass, *Max*	0·05	A-6
vi)	Solubility in water	To pass the test	A-7
vii)	Solubility in alcohol	To pass the test	A-8
viii)	Fineness	As agreed to between buyer and seller	A-9
ix)	Sulphates	To pass the test	A-10

ANNEX A

[*Clause* 3.2, and *Table* 1]

METHODS OF TEST FOR BORIC ACID FOR COSMETIC INDUSTRY

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* 1070 : 1977) shall be used in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF BORIC ACID

A-2.1 Reagents

a) *Mannitol or Sorbitol* — Neutral. The reagent shall satisfy the following test:

> Five grams of the reagent dissolved in 50 ml of carbon dioxide-free water shall require for neutralization not more than 0·3 ml of 0·02 N sodium hydroxide solution using phenolphthalein solution as indicator.

b) *Standard Sodium Hydroxide Solution* — 1 N.

c) *Phenolphthalein Indicator Solution* — Dissolve 0·1 g of phenolphthalein in 60 ml of rectified spirit (*see* IS 323 : 1969) and dilute with water to 100 ml.

A-2.2 Procedure

Accurately weigh about 1 g of the dried material preserved as prescribed in **A-5.2** and dissolve it in about 120 ml of water by heating, avoid boiling Cool the solution to ambient temperature, add approximately 15 g of the mannitol or sorbitol and 0·4 ml of phenolphthalein indicator solution. Titrate the solution with standard sodium hydroxide solution to a distinct pink colour.

NOTE — To ensure that the correct titration end point is obtained, the following standard colour matching solution may be used for comparison with the solution being titrated.

Mix 50 ml of a 3·81 g/l solution of disodium tetraborate ($Na_2B_4O_7 \cdot 10H_2O$), 100 ml of water, 2·0 ml of hydrochloric acid (0·25 N) and 0·4 ml of phenolphthalein indicator solution.

Equal volume of this solution and of the titrated liquid shall be compared in similar beakers.

A-2.3 Calculation

$$\text{Boric acid, percent by mass} = \frac{6{\cdot}184\,V}{M}$$

where

V = volume in ml of standard sodium hydroxide solution used in the titration; and

M = mass in g of the dried material taken for the test.

A-3 TEST FOR ARSENIC

A-3.1 Preparation of Solution

Dissolve 1·000 g of the material in 20 ml of water.

A-3.2 Carry out the test for arsenic as prescribed in IS 2088 : 1983 with the solution obtained in **A-3.1**, using 0·01 mg of arsenic trioxide for comparison.

A-4 TEST FOR HEAVY METALS

A-4.1 Apparatus

a) *Nessler Cylinders* — 50 ml capacity.

A-4.2 Reagents

a) *Dilute Hydrochloric Acid* — approximately 1 N.

b) *Hydrogen Sulphide Solution* — freshly prepared, saturated solution.

c) *Standard Lead Solution* — Dissolve 1·600 g of lead nitrate in 100 ml of water to which 1 ml of nitric acid has been added. Make the volume to one litre. Dilute 10 ml of this solution again to one litre. One millilitre of this solution is equivalent to 0·01 mg of lead (as Pb).

A-4.3 Procedure

Dissolve 1·000 g of the material in 25 ml of hot water, and 0·5 ml of dilute hydrochloric acid, heat to 80°C, transfer to a Nessler cylinder and add 10 ml of hydrogen sulphide solution. Make up the solution to 50 ml. Carry out a control test using 2 ml of standard lead solution and the same quantity of reagents in the same total volume of the reaction mixture. Compare the colour of the solutions 10 minutes after the addition of hydrogen sulphide solution.

A-4.3.1 The prescribed limit shall be taken as not having been exceeded if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

A-5 LOSS ON DRYING

A-5.1 Apparatus

a) *Dish* — Circular, made of glass or aluminium, 100 mm in diameter and 10 mm deep, with tight-fitting cover or lid.

A-5.2 Procedure

Clean, dry and weigh the dish and cover. Weigh accurately about 10 g of the material as quickly as possible, the final mass being taken with the dish covered by the lid. Spread the boric acid evenly in the dish, by gently tapping and shaking. Keep the dish with the lid off, in an oven at 40 ± 2°C for 4 hours. At the end of the period transfer the dish to a vacuum desiccator and connect the desiccator to the pump. The final pressure inside the desiccator, when steady conditions have been attained, should be not more than 200 mm Hg. After the minimum pressure has been reached, leave the desiccator connected to the vacuum for one hour. Disconnect the desiccator from the vacuum line and allow to stand under vacuum at room temperature for 12 hours. Break the vacuum by admitting dry air through a guard tube containing fused calcium chloride. Weigh the dish with cover on. Use the dried material for test in **A-2**.

A-5.3 Calculation

$$\text{Loss on drying, percent by mass} = \frac{100\,m}{M}$$

where

m = loss in mass in g on drying; and

M = mass in g of the material taken for the test.

A-6 TEST FOR IRON

A-6.1 Reagents

a) *Concentrated Hydrochloric Acid* — *see* IS 265 : 1976.

b) *Ammonium Persulphate* — solid.

c) *Butanolic Potassium Thiocyanate Solution* — Dissolve 10 g of potassium thiocyanate in 10 ml of water and add sufficient n-butanol to make up the volume to 100 ml. Shake vigorously until the solution is clear.

d) *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [$FeSO_4.(NH_4)_2.SO_4.6H_2O$] in water in a 1-litre flask, add 4 ml of concentrated sulphuric acid (*see* IS 266 : 1977) and dilute with water to the mark. When required for test, dilute 10 ml of this solution to 100 ml in a graduated flask. One millilitre of the diluted solution contains 0·01 mg of iron (as Fe).

A-6.2 Procedure

Dissolve 1·000 g of the material in hot water and make up the volume to 100 ml. Take 10 ml of the solution and add 1 ml of concentrated hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake vigorously and allow the layers to separate. Carry out a control test with 5 ml standard iron solution in place of the material and the same quantities of other reagents. Compare the colour of the butanol layers.

A-6.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour of the butanol layer in the test with the material is not greater than that of the colour produced in the control test.

A-7 TEST FOR SOLUBILITY IN WATER

A-7.1 Procedure

Take 25 ml of water maintained at a temperature of 27 ± 1°C and add to it 1·000 g of the material. Shake thoroughly.

A-7.1.1 The material shall be taken to have passed the test if the solution obtained is clear.

A-8 TEST FOR SOLUBILITY IN ALCOHOL

A-8.1 Reagent

a) *Rectified Spirit* — *see* IS 323 : 1959.

A-8.2 Procedure

Heat 2·000 g of the material with 20 ml of rectified spirit under reflux condenser.

A-8.2.1 The material shall be taken to have passed the test if the solution, while hot, is clear with no insoluble residue.

A-9 DETERMINATION OF SIEVE ANALYSIS

A-9.1 Apparatus

a) *Test Sieve* — Specified IS sieve (*see* IS 460), prepared for the test by removing any film, grease or other dirt and then by drying.

A-9.2 Procedure

Weigh about 10·0 g of the material and transfer it to the test sieve. Cover the sieve and screen the material in a Ro-Tap or a similar machine for 20 minutes. Two small square rubber cubes are introduced along with the material on the sieve to facilitate the breaking up of any soft lumps of the caked material or camel brush may be used very softly to break the agglomerates formed. After 20 minutes stop the machine and brush the residue on the sieve into a tared weighing dish. Weigh the dish and determine the mass of the residue.

A-9.3 Calculations

$$\text{Material passing through the specified IS sieve, percent by mass} = \frac{100\,(1-m)}{M}$$

where

m = mass in g of the material retained on the test sieve, and

M = mass in g of the material taken for test.

A-10 DETERMINATION OF SULPHATES

A-10.1 Reagents

a) *Barium Chloride Solution* — 0·5 M. Dissolve 122·1 g of barium chloride ($BaCl_2.2H_2O$) in 1 000 ml of distilled water.

b) *Hydrochloric Acid* — *see* IS 265 : 1976.

c) *Barium Sulphate Reagents* — Mix 15 ml of 0·5 M barium chloride, 55 ml of water and 20 ml of sulphate free alcohol (IS 232 : 1959), add 5 ml of 0·018 1 percent w/v solution of potassium sulphate, dilute to 100 ml with water and mix. Barium sulphate reagent must be freshly prepared.

A-10.2 Procedure

Boil 3 g of material in 30 ml of water and add 1 ml of hydrochloric acid. Cool and filter. Transfer to a Nessler's cylinder. Dilute to 45 ml with water and add 5 ml of barium sulphate reagent. Stir the solution immediately with a glass rod and allow to stand for five minutes.

For control test, in a separate Nessler's cylinder, take 1·0 ml of 0·261 4 percent w/v solution of potassium sulphate and 1 ml of hydrochloric acid, dilute to 45 ml with water and add 5 ml of barium sulphate reagent. Stir immediately with a glass rod and allow to stand for five minutes.

The sample is taken to have passed the test if the turbidity produced in sample is not greater than in control test.

ANNEX B

(*Clause* 5.1)

SAMPLING OF BORIC ACID

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container shall be mixed thoroughly by suitable means.

B-1.5 The sample shall be placed in suitable, clean and dry glass containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled with the sample.

B-1.7 Each sample container shall be sealed airtight after filling and marked with full details of sampling, date of sampling and year of manufacture of the material.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers of the same grade in a single consignment of the material, drawn from the same batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture or of different types of containers, the containers of the same type belonging to the same batch shall be grouped together and each such group shall constitute a lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of this specification.

B-2.2 The number of containers (n) to be chosen from the lot shall depend on the size of the lot (N) and shall be as specified in Table 2.

B-2.3 The containers shall be chosen at random from the lot and, in order to ensure randomness of selection, a random number, table (*see* IS 4905 : 1968) as agreed to between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure shall be adopted.

> 'Starting from any container, count as 1, 2, 3 . . . . . . , etc, up to r and so on where r is equal to the integral part of N/n. Every rth container thus counted shall be taken out to give a sample for test.'

Table 2 Number of Containers to be Selected for Sampling

(*Clause* B-2.2)

Lot Size (N)	No. of Containers to be Selected (n)
(1)	(2)
Up to 4	Each container
5 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 to 1 000	7
1 001 and above	8

B-3 TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected (Table 2). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given in Table 1, column 2 and shall be not less than 500 g.

B-3.1.2 *Thoroughly mix* all portions of the material drawn from the same container. Out of these portions, equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 500. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.1.3 The remaining portion of the material from each container (after the quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 50 g. These parts shall be immediately transferred to thoroughly dried bottles which are than sealed airtight with stoppers and labelled with all the particulars of sampling given under **B-1.7**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

B-3.2 Referee Sample

The referee sample shall consist of the composite sample (**B-3.1.2**) and a set of individual samples (**B-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of boric acid shall be conducted on each of the individual samples.

B-4.2 Tests for the remaining characteristics shall be conducted on the composite samples.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

The mean (X) and range (R) of the test results for boric acid shall be calculated (range being the difference between the maximum and minimum values of the test results).

B-5.1.1 The lot shall be declared to have satisfied the requirement for boric acid if the value of the expression ($X - 0{\cdot}6R$) is equal to or greater than the value specified in Table 1.

B-5.2 For Composite Sample

The test results on the composite sample shall meet the corresponding requirements specified in Table 1.

B-5.3 A lot shall be declared as conforming to this specification if it satisfies the relevant requirements for each of the characteristics listed in **3** and in Table 1. If the requirements for any of the characteristics are not met, the lot shall be declared to have not satisfied the requirements of the specification.

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